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2. Patent application number *(The Patent Office will fill in this part)* — 0223126.4

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

JOHNSON MATTHEY PUBLIC LIMITED COMPANY
2-4 COCKSPUR STREET
TRAFALGAR SQUARE
LONDON SW1 5BQ

Patents ADP number *(if you know it)* — 536268007

If the applicant is a corporate body, give the country/state of its incorporation — GB

4. Title of the invention — ENGINE EXHAUST GAS TREATMENT

5. Name of your agent *(if you have one)* — ANDREW DOMINIC NUNN

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

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Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

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Number of earlier application	Date of filing *(day / month / year)*

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Claim(s)	3
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11. I/We request the grant of a patent on the basis of this application.

Signature [signature] Date 4/10/02

A D NUNN

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ENGINE EXHAUST GAS TREATMENT

This invention relates to engine exhaust gas treatment, and in particular to a system and process applied to a lean-burn engine.

The exhaust of such engines commonly contains CO, CO_2, HC, oxygen, nitrogen and steam, and the atmospheric pollutants NOx and particulates. Engine designs have evolved to limit the total and relative concentrations of the pollutants and also for such objectives as limiting noise. Commercially available diesel engine fuels have changed in the direction of lower sulfur content, broadly from about 2500 ppm in the 1970s, to 500ppm in the 1980s, 50ppm in the early 1990s and now under 10ppm. Whereas treatment measures effective in steady running conditions (hot exhaust) have become available, emissions during idling or cold-start (cool exhaust) continue to present problems. One such problem is the emission of NO_2 in cool exhaust conditions, which appears to have increased in recent years.

According to the invention an exhaust treatment system for a lean-burn internal combustion engine capable of operating in a first mode producing relatively cool exhaust gas and in a second mode producing a relatively hot exhaust gas and comprising a regenerable NO_x absorber is characterised in that the NO_x absorber comprises an upstream zone and a downstream zone, the upstream zone being substantially inactive in said cool gas for the reaction of HC with NO_2 to give NO.

This system corresponds to a process of operating a lean-burn internal combustion engine operable in a first mode producing a relatively cool exhaust gas and a second mode producing a relatively hot exhaust gas, said process comprising:

(a) in said first mode absorbing NOx from the flowing gas in a first charge of regenerable absorbent, said first charge being substantially inactive for catalysing the reaction of HC with NO_2 at the prevailing temperature and passing the resulting gas to a second charge of regenerable absorbent; and

(b) in said second mode absorbing said NO_x from the flowing gas and in said first charge and in said second charge.

The process comprises also periodically regenerating said charges.

The first mode, producing the relatively cool exhaust gas, corresponds for example to cold-start, idling at regular or chance stopping places, and general coasting or low-load running. The temperature of the gas corresponds to an NO_2-rich state of the equilibrium:

$$NO + \frac{1}{2} O_2 \rightarrow NO_2$$

Typical temperatures are in the range 120-200°C, especially 140-190°C. Although the gas contains NO and O_2, the forward reaction, even over a platinum catalyst, is slow and would not make up the content of NO_2 if the NO_2/HC reaction were catalysed.

The second mode, producing the relatively hot exhaust gas, corresponds for example to steady running at over 1000 rpm, such as in open country, mild hill climbing and between delivery/collection stops. It occurs especially in acceleration from the first mode. The temperature of the gas preferably corresponds to a rate of the reaction over the oxidation catalyst (if used) and elsewhere:

$$NO + \frac{1}{2}O_2 \rightarrow NO_2$$

sufficient to provide NO_2 at least equal to NO. This ensures that NO_x is efficiently absorbed in the downstream zone and also that the two absorption zones become rapidly regenerated to the extent required. Typical temperatures are over 200°C, especially over 250°C, for example 300-600°C.

Whereas NO_x absorbers commonly contain catalyst for regeneration with reductant or NO_x-specific reactant, some of such catalysts also promote the reaction of HC with NO_2. For this reason the upstream zone is substantially ineffective for that reaction at the temperatures of the first mode. In particular it is preferably free of platinum and related metals of like activity. It may contain catalyst at a concentration too low for effectiveness at such temperatures, but sufficient at temperatures of the second

mode. It may contain catalyst ineffective at first mode temperatures as the result of poisoning but effective at second mode temperatures. The upstream zone may contain catalyst effective, at least at second mode temperatures, for reaction of sorbed or gaseous NO_x with reductant or NO_x-specific reactant. It may include for example Rh.

The effect of the upstream zone is to pass to the downstream zone a gas having an NO_2/NO ratio higher than if the NO_2+HC reaction had been substantial. Such a ratio of at least 1:1 is necessary for absorption by basic materials, to give nitrites and nitrates, NO alone being not so absorbed. To effect a further increase in that ratio the system preferably includes a NO oxidation catalyst between the zones. The NO_2-maintaining effect of the upstream zone according to the invention is especially valuable since usually the catalytic NO oxidation is slow at cool exhaust temperatures. The oxidation catalyst preferably effects also the conversion of CO and HC to CO_2 and steam. It may be in two successive parts, optimised respectively for CO/HC oxidation and NO oxidation.

If the gas to be treated contains particulate it may be filtered before discharge to atmosphere. If the particulate contains soot, a mixture of carbon and hydrocarbons in various proportions, the filter should be disposed between the NO oxidation catalyst and the NOx absorber.

The filter may be any capable of trapping the soot without causing excessive back-pressure. In general, ceramic, sintered metal or woven or non-woven wire filters are usable, and wall-flow honeycomb structures may be particularly suitable. The structural material of the filter is preferably porous ceramic oxide, silicon carbide or sintered metal. A coating such as alumina, and also a catalyst such as one or more PGMs (e.g. Pt with MgO) or La/Cs/V_2O_5 may be present. The soot is generally carbon and/or heavy hydrocarbons, and is converted to carbon oxides and H_2O. Certain embodiments of this principle are in commercial use in Johnson Matthey's Continuously Regenerating Trap technology, and are described in the above-mentioned EP-A-0341832 and US patent no. 4902487, the teaching of which is incorporated herein by reference.

The catalysts and absorbent are suitably supported on a ceramic or metal honeycomb, the ceramic comprising one or more of alumina, silica, titania, cordierite, ceria, zirconia, silicon carbide or other, generally oxidic, material. The honeycomb

carries a washcoat and, in one or more layers thereon, the active catalytic and/or absorptive material, to be described in more detail below. The honeycomb has typically at least 50, for example 50-400, cells per square inch (cpsi), possibly more, e.g. up to 800, or up to 1200 if composed structurally of metal. Generally the range 200-800 is preferred for the catalysts and absorbent. Two or more of the agents for catalysis, absorption and filtering may be supported on a single honeycomb unit, especially as described in our co-pending application PCT/GB00/03064.

In the oxidation catalyst the active material comprises generally a platinum group metal ("PGM"), especially platinum and/or palladium, optionally with other PGMs, e.g. rhodium, and other catalytic or promoting components. The exact compositions and structure of the oxidation catalyst are not critical to operation of the invention, and hence may be varied according to the requirements of the situation. A low temperature light-off formulation is generally preferred. Conventional manufacturing techniques may be used. The catalyst should of course be sized and composed to achieve the necessary conversions, and the design should minimise trapping of soot within its honeycomb.

The NO_x absorbent (referred to also as a 'NO_x-trap'), to be described further below, may be provided in one unit or a succession of separate units. It may be in the form of active layers on a conventional honeycomb substrate, or may be in the form of serial deposits on a single honeycomb or possibly multiple honeycombs. If a catalyst system is associated with the absorbent, that is, the absorber is 'catalysed', the catalytic material may be for example co-precipitated or co-impregnated or co-deposited with NOx absorbent or present as one or more sandwiched layers or as fine (e.g. 10-500 microns) particles on or in a layer of absorbent or among particles of absorbent.

The absorbent may be selected from:

(a) solid basic materials for example compounds of alkali metals, alkaline earth metals, rare earth metals and transition metals, capable of forming nitrates and/or nitrites of adequate stability in absorbing conditions and of evolving nitrogen oxides and/or nitrogen in regenerating conditions; and/or (b) adsorptive materials such as zeolites, carbons and high-area oxides.

Compounds (a) may be present (before NOx absorption) as composite oxides, e.g. of alkaline earth metal and copper such as Ba-Cu-O or MnO_2-$BaCuO_2$, possibly with added Ce oxide, or Y-Ba-Cu-O and Y-Sr-Co-O. (The oxides are referred to for simplicity, but in practice hydroxides, carbonates and nitrates are present, depending on

the temperature and gas composition). Whichever compounds are used there may be present - except as herein described for the differing requirements of the upstream zone and downstream zone - one or more catalytic agents, such as precious metals, effective for such reactions as the interchanges of the nitrogen oxides and the action of reductant and/or NOx-specific reactant.

For regeneration of the NO_x absorber:

1. in a simple process higher temperature suffices. The temperature should preferably be high enough to ensure a preponderance of NO in the outlet gas. This is usable if local regulations permit emission of NO, at least in open conditions wherein it can disperse without causing offence. A process of this type is described in our co-pending application published as WO 01/94760.

2. a non-selective regenerant (reductant), hydrocarbon may be introduced, for example gasoline or diesel fuel or another such as light oil, kerosene or a C_3 to C_8 paraffin; Conveniently reductant is introduced by temporary rich operation of the engine An alternative reductant is hydrogen (suitably generated <u>in situ</u> on board the vehicle) or a readily dehydrogenatable reductant such as a lower alcohol, especially methanol or ethanol.

3. a NO_x-specific reactant may be used, especially a nitrogen hydride for example ammonia or hydrazine. This can be injected as such or as a solution in e.g. water or as a precursor compound, for example urea or aqueous urea solution, producing the reactant in exhaust treatment conditions. Such compounds are referred to herein as 'ammonia' at stages after injection. Injectors for such reactants or compounds, possibly using carrier gas such as air, have been published. For regeneration using a NO_x-specific reactant, the oxidant level can be decreased less, if at all, than when using reductant. Indeed the reactant may be used especially in lean conditions, for example:

(a) exhaust gas as generated by the engine or as issuing from a preceding step of exhaust treatment;

(b) such gas to which reductant short of equivalence has been or is being added;

(c) gas made leaner, for example when the reactant is injected with the aid of air.

Regeneration using NO_x-specific reactant is also effective in:

(d) rich or equivalent gas and also in gas into which reductant has been introduced e.g. to provide for reaction increasing gas temperature but leaving the gas net-lean in composition.

If injection of the reductant or reactant is used, this may, for mechanical simplicity, be upstream of one or more stages upstream of the second NO_x absorber, provided unwanted reaction is avoided by careful temperature control or by bypassing through a non-reactive zone of such stage(s).

Usually the regeneration phase can be a small fraction, e.g. 0.1% to 5%, of engine running time, depending of course on operating conditions.

When the regenerant is 'non-selective', it is normally used in excess over the NOx to be reacted. This results in a short emission of reductant to atmosphere. To limit or prevent such emission, the exhaust containing the effluent of regeneration is contacted with a final oxidation catalyst comprising oxygen storage material. Such material supplies oxygen during the regeneration, but absorbs oxygen between regenerations.

The starting exhaust gas preferably contains less than 20ppm SO_2 v/v, measured on complete, that is, steam-containing, exhaust gas. This corresponds to a sulfur content of under 500ppm as S in engine fuel for a typical air/fuel ratio of 30:1. Preferably the S content of the engine fuel used is less than 50ppm, especially less than 10ppm. If fuel of higher S content is to be used, the upstream zone may contain a pre-bed of SOx absorbent or a sacrificial inlet sub-zone. Such pre-bed or sacrificial material may be periodically replaced or regenerated by known procedures.

The invention provides an engine equipped with the exhaust treatment system and a process for operating it. The engine may be for example a diesel or GDI. If diesel, it may be for heavy duty, for example in an omnibus, river bus, delivery van or railway locomotive or railcar. Very usefully it is an existing engine modified by addition of the defined system, thus extending its useful life into a period of more rigorous emission regulation: such an engine should preferably be cleansed of sulphurous residues before adding the system, then operated on very low sulfur fuel. However, an important application of the invention is to modern engines for moderate or light duty, especially those designed for low NO_x emission and/or low noise especially those producing exhaust gas of relatively high NO_2:NO ratio.

The oxygen storage material typically comprises an oxide of a metal capable of exercising 2 or more valency levels. One class of such materials is based on ceria, examples comprising also zirconia and/or being doped with PGM. Another class of such materials is based on manganese oxides, for example in combination with zirconia as described in our co-pending application published as WO 99/34904. A combination of phase-distinct oxides in the atomic ratio 50:50 to 70:30 Mn:Zr, with 2%w/w Pd doping, shows high activity for taking oxygen in and liberating it to CO oxidation, according to a standard test.

The oxygen storage material may be used in association with catalyst for oxidation of regenerant, suitably in a structure analogous to those mentioned above for catalysed absorbers.

The invention is illustrated by the accompanying drawings in which:

Figure 1 is a block diagram of the system as applied to a diesel engine; and

Figure 2 is a block diagram of the system as applied to a GDI gasoline engine,

Referring to Fig. 1, the exhaust gas of 1.9 litre turbocharged diesel engine 10 is fed to NOx absorber 12 constituting the upstream zone of the total NOx absorber of the system. Absorber 12 consists of a 400 cpsi cordierite honeycomb carrying an active alumina washcoat containing magnesium oxide and 0.1%w/w of rhodium. The product of absorber 12 is fed to oxidation catalyst 14, which consists of a wash-coated honeycomb similar to that of 12 but containing platinum. For some conditions 14 can be omitted. The product of 14 is fed to NOx absorber 16, which is similar to 12, but contains barium oxide and platinum instead of magnesium oxide and rhodium. The product of 16 is fed to final oxidation catalyst 18, which consists of a wash-coated honeycomb similar to 12 but containing platinum as catalyst and 50:50 (atomic %) Mn:Zr oxides as oxygen storage material.

In its first mode of operation, such as in idling, the exhaust gas is typically at 150 °C and contains NO and NO_2. The NO_2 is absorbed in 12 with no catalysis of the HC/NO_2 reaction. The resulting gas passes through oxidation catalyst 14 with incomplete if any oxidation of NO to NO_2 owing to the low temperature and then into absorber 16, in which further NOx is absorbed. Owing to absorption at 12 and to the generally low NOx engine output at idling, not much NOx is finally emitted, and that is mainly NO, which is odourless and becomes well dispersed into the atmosphere before smellable amounts of NO_2 are formed.

In the second mode of operation, such as on open road, the exhaust gas is typically at 300°C and contains NO and NO_2 at a higher concentration than in the first mode. Some NO_x is absorbed at 12. If the NO_2 concentration is at least equivalent to that of NO and the NO_2/HC reaction is not present, the gas leaving 12 can pass direct to 16 without the need for 14. In a more versatile process the gas leaving 12 passes into oxidation catalyst 14, which is active enough to oxidise NO to at least equivalent NO_2. The resulting NOx mixture is absorbed at 16 until the capacity limit of 16 is attained. If no oxidation catalyst 14 has been used, the NO_x-free gas from 16 contains CO and HC. These are removed over final oxidation catalyst 18 by reaction with oxygen present in the gas.

The second mode of operation also includes regeneration of 16. If this involves intermittent introduction of reductant, for example by adjustment of the engine to rich operation, the gas leaving 16 contains HC and CO temporarily in excess of oxygen. To meet this situation, catalyst 18 contains an oxygen storage material, which accumulates oxygen during running and releases it during regeneration of absorbers 12 and 16, to oxidise the said HC and CO. If regeneration involves injection of NO_x-specific reactant, catalyst 18 should have SCR activity and should not undergo thorough regeneration, to limit ammonia emission. If NO emission is permitted, regeneration can be thermal only.

Referring to Fig. 2, the exhaust gas of a 2.0 litre GDI spark-ignition gasoline engine 20 is fed to TWC 22 which also acts a NOx absorber constituting the upstream zone of the total NO_x absorber of the system. TWC 22 consists of a 400 cpsi cordierite honeycomb carrying an active alumina washcoat containing 0.1%w/w of rhodium; it is

chosen to be inactive for the NO_2/HC reaction in cool exhaust. The product of TWC 22 is fed to conventional NO_x absorber 24, which contains barium oxide and platinum. The product of 24 is fed to final oxidation catalyst 26 which consists of a wash-coated honeycomb similar to 22 but containing platinum as catalyst and 50:50 (atomic %) Mn:Zr oxides as oxygen storage material. If emission regulations permit, 26 can be omitted.

In the first mode of operation of Fig 2, such as in idling, the exhaust gas is typically at 150°C and contains NO and NO_2. The NO_2 is absorbed in TWC/absorber 22 with no catalysis of the HC/NO_2 reaction. The resulting gas passes into absorber 24, in which further NO_x is absorbed. Owing to absorption at 22 and to the generally low NOx engine output at idling, not much NO_x is finally emitted, and that is mainly NO, which is odourless and becomes well dispersed into the atmosphere before smellable amounts of NO_2 are formed.

In the second mode of operation, of Fig.2, such as on open road, the exhaust gas is typically at 300°C and contains NO and NO_2 at a higher concentration than in the first mode. Some NO_x is absorbed in TWC 22. The NO_2:NO concentration ratio increases over TWC 22 . The resulting NO_x mixture is absorbed at 24 until the capacity limit of 24 is attained. If the NO_x-free gas from 24 contains CO and HC. These are removed over final oxidation catalyst 26 by reaction with oxygen present in the gas.

The second mode of operation of Fig.2 includes regeneration of 24. If this involves intermittent introduction of reductant, for example by adjustment of the engine to rich operation, the gas leaving 24 contains HC and CO temporarily in excess of oxygen. To meet this situation, catalyst 26 contains an oxygen storage material, which accumulates oxygen during running and releases it during regeneration of absorbers 22 and 24, to oxidise the said HC and CO. If regeneration involves injection of NOx-specific reactant, catalyst 24 should have SCR activity.

CLAIMS:

1. An exhaust treatment system for a lean-burn internal combustion engine capable of operating in a first mode producing relatively cool exhaust gas and in a second mode producing a relatively hot exhaust gas and comprising a: regenerable NO_x absorber, characterised in that the NO_x absorber comprises an upstream zone and a downstream zone, the upstream zone being substantially inactive in said cool gas for the reaction of HC with NO_2 to give NO.

2. A process of operating a lean-burn reciprocating internal combustion engine operable in a first mode producing a relatively cool exhaust gas and a second mode producing a relatively hot exhaust gas, said process comprising:
   (a) in said first mode absorbing NO_x from the flowing gas in a first charge of regenerable absorbent, said first charge being substantially inactive for catalysing the reaction of HC with NO_2 at the prevailing temperature and passing the resulting gas to a second charge of regenerable absorbent; and
   (b) in said second mode absorbing said NO_x from the flowing gas and in said first charge and in said second charge.

3. A system or process according to claim 1 or 2, wherein the temperature of the gas in the first mode corresponds to an NO_2-rich state of the equilibrium:

$$NO + \tfrac{1}{2}O_2 \rightarrow NO_2.$$

4. A system or process according to claim 3, wherein the temperature is in the range 120-200°C, especially 140-190°C.

5. A system or process according to any preceding claim, wherein the temperature of the gas in the second mode corresponds to a rate of the reaction:

$$NO + \tfrac{1}{2}O_2 \rightarrow NO_2$$

sufficient to provide NO_2 at least equal to NO.

6. A system or process according to claim 5, wherein the temperatures over 200°C, especially over 250°C, for example 300-600°C.

7. A system or process according to any preceding claim, wherein the first mode absorber is ineffective for the NO_2/HC reaction in virtue of: freedom from platinum and metals of like activity; and/or containing catalyst at a concentration too low for effectiveness at first mode temperatures, but sufficient at temperatures of the second mode; and/or containing catalyst ineffective at first mode temperatures as the result of poisoning but effective at second mode temperatures.

8. A system or process according to any preceding claim, including an NO oxidation catalyst between the zones.

9. A system or process according to claim 8, wherein a soot filter is disposed between the NO oxidation catalyst and the NO_x absorber.

10. A system or process according to any preceding claim, wherein regeneration of the NO_x absorber(s) is by a non-selective reductant used in excess over the NO_x to be reacted, and emission of reductant to atmosphere is limited or prevented by contacting the exhaust containing the effluent of regeneration with a final oxidation catalyst comprising oxygen storage material.

11. A system or process according to any preceding claim, wherein the starting exhaust gas preferably contains less than 20ppm SO_2 v/v, measured on complete, that is, steam-containing, exhaust gas.

12. A system or process according to claim 11, wherein the exhaust gas is the product of an engine fed with fuel of sulfur content of under 500ppm as S, especially less than 50ppm, more especially less than 10ppm.

13. A monolith comprising a first NO_x-trap composition in a first zone and a second NO_x-trap composition in a second zone, the first NO_x-trap composition being free of platinum or contains platinum and a catalyst poison for limiting the activity of the platinum to catalyse the reaction of HC with NO_2 to give NO at relatively low exhaust gas temperatures and the second NO_x-trap composition comprising at least one PGM,

whereby the first NO_x-trap composition is substantially inactive for the reaction of HC with NO_2 to give NO in relatively cool exhaust gas.

14. A diesel engine or lean-burn gasoline/LPG engine including exhaust treatment according to any preceding claim.

15. A system or process, substantially as described herein with reference to the accompanying drawings.

Fig. 1

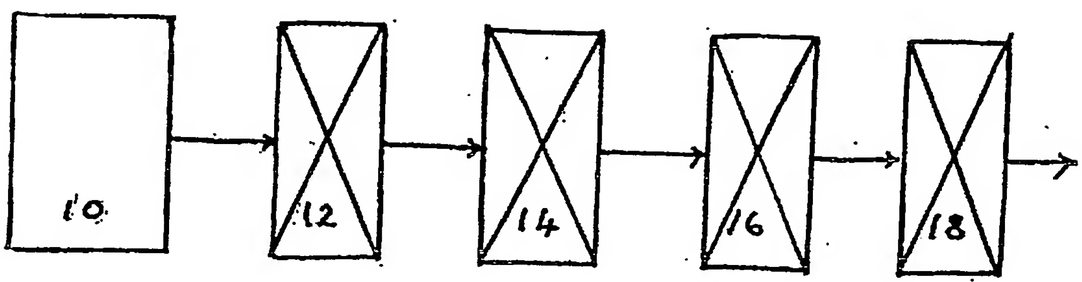
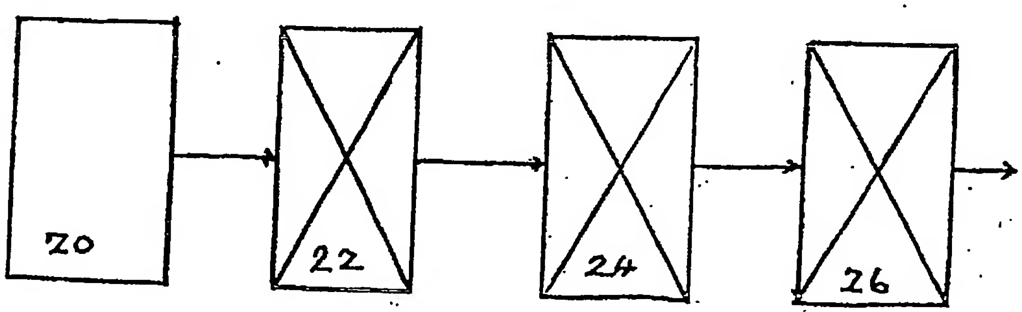


Fig. 2



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